CLAIMS

What is claimed is:

10

15

20

Claim 1. A process for the production of biodiesel from a lipid feedstock comprising a plurality of free fatty acids, said process comprising:

introducing the lipid feedstock to a conditioning reactor;

conditioning the lipid feedstock in the conditioning reactor, wherein the lipid feedstock is heated to a temperature in the range of about 55°C to about 65°C, mixed by an agitator with a power input per unit volume of about 0.5 watts per gallon (W/gal) to about 1.5 W/gal and filtered using a rotary screen to remove solids having a dimension over about 1 micron to about 200 microns to produce a conditioned lipid feedstock;

measuring the concentration of free fatty acids in the conditioned lipid feedstock; reacting the plurality of free fatty acids in the conditioned lipid feedstock with a purified glycerin product in a glycerolysis reactor, wherein the plurality of free fatty acids in the feedstock is mixed with the purified glycerin product using an agitator with a power input per unit volume of about 5.5 W/gal to about 60 W/gal and continuously reacted with the purified glycerin product in the absence of a catalyst at a temperature of about 180°C to about 250°C and at a pressure of about 0.1 pounds per square inch absolute to about 7 pounds per square inch absolute in a glycerolysis reaction to produce a glycerolysis reactor effluent stream that contains less than 0.5 percent by weight of free fatty acids and a plurality of glycerides, the purified glycerin product being continuously added to the glycerolysis reactor at a rate in the range of about 110 percent to about 400 percent of the stoichiometric amount of glycerin required for the

glycerolysis reaction, water being continuously removed from the glycerolysis reactor as a vapor through a fractionation column that returns condensed glycerin to the glycerolysis reactor, said glycerolysis reactor comprising at least two continuous stirred tank reactors that are operated in series; said reactors having a combined residence time of not more than about 500 minutes;

reacting the plurality of glycerides contained in the glycerolysis effluent stream with a purified methanol product comprising methanol in a transesterification reactor, wherein the plurality of glycerides is mixed with said purified methanol product and potassium hydroxide using an agitator with a power input per unit volume of about 3.0 W/gal to about 50 W/gal and continuously reacted with the methanol at a temperature in the range of about 25°C to about 65°C and at a pressure of about 1 bar in an alkali catalyzed transesterification reaction to produce a transesterification reactor effluent stream that contains a plurality of fatty acid methyl esters and glycerin, the purified methanol product being added to the transesterification reactor at a rate equal to about 200 percent of the stoichiometric amount of methanol required for the alkali catalyzed transesterification reaction, the potassium hydroxide being added to the transesterification reactor at a rate of about 0.5 percent by weight to 2.0 percent by weight of glycerides present in the glycerolysis effluent stream, said transesterification reactor comprising two continuous stirred tank reactors that are operated in series, said reactors having a combined residence time of about 15 minutes to about 90 minutes;

separating the plurality of fatty acid methyl esters from the glycerin in the transesterification effluent stream in a continuous clarifier, wherein a first liquid phase in which the plurality of fatty acid methyl esters are concentrated and a second liquid phase in which glycerin is concentrated are continuously separated at a temperature of about 25°C to about 65°C to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

purifying the fatty acid methyl ester rich stream in a fatty acid methyl ester distillation column and recovering the methanol from the fatty acid methyl ester rich stream to produce a purified biodiesel product and a first wet methanol stream, wherein the fatty acid methyl ester distillation column is operated at a temperature in the range of about 180°C to about 230°C and at a pressure in the range of about 0.1 pounds per square inch absolute to about 2 pounds per square inch absolute;

5

10

15

20

purifying the glycerin rich stream and recovering the methanol from the glycerin rich stream to produce the purified glycerin product and a second wet methanol stream, wherein the potassium hydroxide in the glycerin rich stream is reacted with phosphoric acid to produce an insoluble salt having fertilizer value that is removed from the glycerin rich stream in a solids separation operation and thereafter rinsed with methanol and filtered, the pH of the glycerin rich stream being adjusted to neutral by adding an alkali solution and the glycerin rich stream being further purified in a glycerin distillation column that is operated at a temperature in the range of about 180°C to about 230°C and at a pressure in the range of about 0.1 pounds per square inch absolute to about 2 pounds per square inch absolute and in a decolorization column comprising a packed bed of activated carbon column operated at a temperature in the range of about 40°C to about 200°C;

purifying the wet methanol streams by removing water to produce a purified methanol product, wherein the wet methanol streams are purified in a methanol distillation column that is operated at a temperature in the range of about 60°C to about 110°C and at a pressure in the range of about 14 pounds per square inch absolute to about 20 pounds per square inch absolute;

recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the plurality of free fatty acids; and

recycling at least a portion of the purified methanol product into the transesterification reactor for reaction with the plurality of glycerides.

Claim 2. A process for the production of biodiesel from a lipid feedstock comprising a free fatty acid, said process comprising:

5

10

15

20

conditioning the lipid feedstock in a conditioning reactor, wherein the lipid feedstock is heated, mixed and filtered to produce a conditioned lipid feedstock;

reacting the free fatty acid in the conditioned lipid feedstock with a purified glycerin product in a glycerolysis reactor, wherein the free fatty acid in the feedstock is mixed and continuously reacted with the purified glycerin product in the absence of a catalyst at an appropriate temperature and pressure in a glycerolysis reaction to produce a glycerolysis reactor effluent stream that contains a glyceride, the purified glycerin product being continuously added to the glycerolysis reactor at a rate that is greater than the stoichiometric amount of glycerin required for the glycerolysis reaction, water being continuously removed from the glycerolysis reactor as a vapor;

reacting the glyceride contained in the glycerolysis effluent stream with a purified alcohol product comprising an alcohol in a transesterification reactor, wherein the glyceride is mixed with said purified alcohol product and continuously reacted with the alcohol at an appropriate temperature and pressure in an alkali catalyzed transesterification reaction to produce a transesterification reactor effluent stream that contains a fatty acid alkyl ester and glycerin, the purified alcohol product being added to the transesterification reactor at a rate that is greater than the stoichiometric amount of alcohol required for the alkali catalyzed transesterification reaction;

separating the plurality of fatty acid alkyl esters from the glycerin in the transesterification effluent stream in a continuous operation, wherein a first liquid phase in which the plurality of fatty acid methyl esters are concentrated and a second liquid phase in which glycerin is concentrated are continuously separated to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

purifying the fatty acid alkyl ester rich stream in a fatty acid alkyl ester distillation column or a fatty acid alkyl ester fractionation column and recovering the alcohol from it to produce a purified biodiesel product and a first wet alcohol stream;

purifying the glycerin rich stream and recovering the alcohol from it to produce the purified glycerin product and a second wet alcohol stream, wherein the alkali in the glycerin rich stream is reacted with an acid to produce an insoluble salt that is removed from the glycerin rich stream and thereafter filtered and rinsed with the alcohol, the pH of the glycerin rich stream being adjusted to neutral and the glycerin rich stream being further purified in a glycerin distillation column or a glycerin fractionation column and in a decolorization column; and

purifying the wet alcohol streams by removing excess water to produce a purified alcohol product, wherein the wet alcohol streams are purified in an alcohol distillation column or an alcohol fractionation column.

Claim 3. The process of claim 2 further comprising:

5

10

15

20

recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acid.

Claim 4. The process of claim 2 further comprising:

recycling at least a portion of the purified alcohol product into the transesterification reactor for reaction with the glyceride.

Claim 5. A process for the production of biodiesel from a lipid feedstock comprising a free fatty acid, said process comprising:

a step for continuously conditioning the lipid feedstock to produce a conditioned lipid feedstock;

5

10

15

20

a step for continuously measuring the concentration of the free fatty acid in the conditioned lipid feedstock by means of an in-line free fatty acid titration device that produces a signal;

a step for continuously reacting the free fatty acid in the conditioned lipid feedstock in a glycerolysis reaction, wherein the free fatty acid in the feedstock is reacted with glycerin to produce a glyceride in response to the signal from the in-line free fatty acid titration device:

a step for continuously reacting the glyceride in a transesterification reaction, wherein the glyceride is converted to a fatty acid methyl ester and glycerin via an alkali catalyzed;

a step for continuously separating the fatty acid methyl ester from the glycerin to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

a step for continuously purifying the fatty acid methyl ester rich stream and recovering the methanol from the fatty acid methyl ester rich stream to produce a purified biodiesel product and a first wet methanol stream;

a step for continuously purifying the glycerin rich stream and recovering the methanol from the glycerin rich stream to produce a purified glycerin product and a second wet methanol stream;

a step for continuously purifying the wet methanol streams to produce a purified methanol product; and

a step for recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acid.

5

Claim 6. The process of claim 5 further comprising:

a step for recycling at least a portion of the purified methanol product into the transesterification reactor for reaction with the glyceride.

10

Claim 7. A process for the production of biodiesel from a lipid feedstock comprising a free fatty acid, said process comprising:

introducing a lipid feedstock to a conditioning reactor;

conditioning the lipid feedstock, wherein the lipid feedstock is heated, mixed and filtered to produce a conditioned lipid feedstock;

15

reacting the free fatty acid in the conditioned lipid feedstock in a glycerolysis reactor, wherein the free fatty acid in the feedstock is continuously reacted with a stoichiometric excess of glycerin to produce a glyceride via a glycerolysis reaction;

reacting the glyceride in a transesterification reactor, wherein the glyceride is continuously converted to a fatty acid methyl ester and glycerin via an alkali catalyzed transesterification reaction;

20

separating the fatty acid methyl ester from the glycerin, wherein a first liquid phase in which the fatty acid methyl ester is concentrated and a second liquid phase in which glycerin is

concentrated are continuously separated to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

purifying the fatty acid methyl ester rich stream and recovering the methanol from the fatty acid methyl ester rich stream to produce a purified biodiesel product and a first wet methanol stream;

purifying the glycerin rich stream and recovering the methanol from the glycerin rich stream to produce a purified glycerin product and a second wet methanol stream;

purifying the wet methanol streams by removing water from them to produce a purified methanol product;

recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acid; and

recycling at least a portion of the purified methanol product into the transesterification reactor for reaction with the glyceride.

15 Claim 8. The process of claim 7 wherein the introducing the lipid feedstock step further comprises:

introducing a feedstock that includes

5

10

20

at least one free fatty acid at a concentration in the range of about 3 percent to about 97 percent by weight;

moisture, impurities and unsaponafiable matter at a concentration up to about 5 percent by weight; and

a remainder that includes monoglycerides, diglycerides and/or triglycerides.

Claim 9. The process of claim 7 wherein the conditioning the lipid feedstock step produces a conditioned feedstock that is a substantially uniform mixture of liquid lipids having a temperature in the range of about 35°C to about 250°C.

Claim 10. The process of claim 9 wherein the conditioning the lipid feedstock step produces a conditioned feedstock having a temperature in the range of about 45°C to about 65°C.

Claim 11. The process of claim 7 wherein the conditioning the lipid feedstock step produces a conditioned feedstock that is a substantially free of insoluble solids.

Claim 12. The process of claim 7 wherein the reacting the free fatty acid step further comprises:

combining the free fatty acid with an effective amount of glycerin for an effective amount of time to facilitate the glycerolysis reaction under conditions wherein the free fatty acid and the glycerin come into substantially intimate contact.

Claim 13. The process of claim 12 further wherein a low frequency acoustic transducer is used to mix the free fatty acid and the effective amount of glycerin.

Claim 14. The process of claim 12 wherein the reacting the free fatty acid step further comprises:

performing the glycerolysis reaction at a temperature in the range of about 150°C to about 250°C; and

removing water from the glycerolysis reactor.

10

15

20

Claim 15. The process of claim 14 wherein the water is removed as vapor through a fractionation column or a distillation column that returns condensed glycerin to the glycerolysis reactor.

5

Claim 16. The process of claim 7 wherein the reacting the glyceride step further comprises:

contacting the glyceride with an effective amount of methanol and an effective amount of alkali catalyst under conditions wherein the glyceride, the effective amount of methanol and the effective amount of alkali catalyst come into substantially intimate contact; and

10

wherein the effective amount of the alkali catalyst is selected from the group consisting of

> an effective amount of sodium hydroxide, and an effective amount of potassium hydroxide.

15

Claim 17. The process of claim 16 wherein the reacting the glyceride step further comprises: performing the transesterification reaction at a temperature in the range of about 20°C to about 250°C.

20

Claim 18. The process of claim 17 wherein the reacting the glyceride step further comprises: performing the transesterification reaction at a temperature in the range of about 55°C to about 65°C.

Claim 19. The process of claim 7 wherein the reacting the glyceride step further comprises:

performing the transesterification reaction at an absolute pressure in the range of about 1 bar to about 250 bar.

Claim 20. The process of claim 19 wherein the reacting the glyceride step further comprises: performing the transesterification reaction at an absolute pressure of about 1 bar.

Claim 21. The process of claim 16 further wherein a low frequency acoustic transducer is used to mix the glyceride, the effective amount of methanol and the effective amount of alkali catalyst.

10

5

Claim 22. The process of claim 7 wherein the separating the fatty acid methyl ester from the glycerin step involves using the density difference between the first liquid phase and the second liquid phase to separate them in a continuous operation.

15 Claim 23. The process of claim 7 wherein the purifying the fatty acid methyl ester rich stream step further comprises:

using a fatty acid methyl ester distillation column or a fractionation column to separate the fatty acid methyl ester rich stream into a bottoms fraction, an overhead fraction comprising primarily methanol, and a side stream fraction comprising a fatty acid methyl ester product.

20

Claim 24. The process of claim 23 wherein the bottoms fraction produced by the fatty acid methyl ester distillation column or fractionation column comprises impurities, and

unsaponafiable materials, unreacted monoglycerides, unreacted diglycerides, unreacted triglycerides and fatty acids.

Claim 25. The process of claim 23 wherein the fatty acid methyl ester product produced by the fatty acid methyl ester distillation column meets ASTM specification D 6751-02.

5

10

15

Claim 26. The process of claim 23 wherein the overhead fraction produced by the fatty acid methyl ester distillation column or fractionation column comprises essentially methanol.

Claim 27. The process of claim 23 wherein the fatty acid methyl ester distillation column or fractionation column is operated at a pressure below about 2 pounds per square inch absolute.

Claim 28. The process of claim 27 wherein the fatty acid methyl ester distillation column or fractionation column is operated at a pressure in the range of about 0.1 pounds per square inch absolute to about 2 pounds per square inch absolute.

Claim 29. The process of claim 23 wherein the fatty acid methyl ester distillation column or fractionation column is operated at a temperature in the range of about 180°C to about 280°C.

Claim 30. The process of claim 29 wherein the fatty acid methyl ester distillation column or fractionation column is operated at a temperature in the range of about 180°C to about 230°C.

Claim 31. The process of claim 17 wherein the fatty acid methyl ester distillation column or fractionation column contains a packing material.

Claim 32. The process of claim 7 wherein the purifying the glycerin rich stream and recovering methanol step further comprises:

performing glycerin fractionation, wherein the fractions within the glycerin rich stream are separated by distillation;

performing phase separation, wherein the impurities that co-fractionate with glycerin are removed by immiscibility and differences in density; and

glycerin polishing, wherein other impurities are removed from glycerin.

Claim 33. The process of claim 32 wherein the purifying the glycerin rich stream and recovering methanol step further comprises:

performing alkali catalyst precipitation, wherein the glycerin rich stream is reacted with a mineral acid suitable to form an insoluble salt with the alkali catalyst used in the transesterification reaction, wherein the mineral acid is selected from the group consisting of

sulfuric acid, and

5

10

15

20

phosphoric acid;

performing solids separation, wherein the insoluble salt is removed from the liquid permeate;

performing phase separation, wherein a fatty acid methyl ester rich liquid phase and a glycerin rich liquid phase are separated;

performing pH adjustment, wherein the pH of the glycerin rich stream is adjusted by adding an alkali solution;

performing glycerin fractionation, wherein the glycerin rich stream is purified by means of a glycerin distillation column and methanol is collected for further purification and reuse in the process; and

performing glycerin polishing, wherein colored impurities are removed from the glycerin.

Claim 34. The process of claim 33 wherein the performing glycerin phase pH adjustment step is performed using ion exchange media.

10

15

5

Claim 35. The process of claim 33 wherein the insoluble salt is separated using a rotary vacuum drum filter, a plate and frame press or a belt press.

Claim 36. The process of claim 33 wherein the alkali catalyst and mineral acid used for alkali catalyst precipitation are chosen so that their reaction will produce a byproduct salt having fertilizer value; said byproduct salts are washed free of organic materials with a solvent to produce a purified salt and the purified salt is then dried and the solvent is recovered for reuse in the process.

Claim 37. The process of claim 33 wherein the insoluble salt is washed free of organic impurities with a solvent prior to the performing solids separation step or during the performing solids separation step using filtration equipment.

Claim 38. The process of claim 37 further comprising drying the insoluble salt in a drier under conditions wherein temperature of the drier exceeds the boiling point of the solvent at the operating pressure of the dryer; the dryer is optionally operated under a vacuum to improve the drying; and the drier includes a condenser to recover the solvent for reuse.

5

Claim 39. The process of claim 38 wherein the solvent is methanol.

Claim 40. The process of claim 33 wherein the insoluble salt is further processed for use as a fertilizer by dissolving it in water.

10

Claim 41. The process of claim 33 wherein the performing glycerin fractionation step further comprising:

distilling the neutralized crude glycerin stream to produce a bottoms material, a side stream and an overhead stream.

15

Claim 42. The process of claim 33 wherein the bottoms material contains essentially waste materials; the side stream contains essentially glycerin and trace impurities; and the overhead stream contains essentially water and methanol that is recovered and recycled.

20

Claim 43. The process of claim 33 wherein the glycerin distillation column is operated under a reduced pressure of below about 2 pounds per square inch absolute.

Claim 44. The process of claim 43 wherein the glycerin distillation column contains packing material and is operated under a reduced pressure of between about 0.1 and about 2 pounds per square inch absolute.

Claim 45. The process of claim 33 wherein the glycerin distillation column is operated at an elevated temperature between about 180°C and about 280°C.

Claim 46. The process of claim 45 wherein the glycerin distillation column contains packing material and is operated at an elevated temperature between about 180°C and about 230°C.

Claim 47. The process of claim 33 wherein the glycerin polishing step comprises contacting the glycerin with activated carbon at a temperature that is between about 35°C and 200°C.

Claim 48. The process of claim 47 wherein the glycerin polishing step comprises contacting the glycerin with a packed bed of activated carbon for a contact time of less than four hours at a temperature that is between about 40°C and 100°C and wherein activated carbon fines carried through the packed bed are removed by filtration through a hydrophilic filter material.

Claim 49. A process for production of biodiesel and glycerin comprising:

10

15

20

inputting to a glycerolysis reactor an effective amount of glycerin and a feedstock comprising 3 to 100 percent free fatty acids and, optionally, a fat and/or an oil;

reacting in the glycerolysis reactor the glycerin and at least a portion of the feedstock in an esterification reaction, with removal of water, to continuously produce a first intermediate

product comprising glycerides (including monoglycerides, diglycerides and/or triglycerides) and essentially no water;

performing in a transesterification reactor continuous base-catalyzed transesterification of the intermediate product to produce a second intermediate product comprising fatty acid methyl esters and glycerin;

continuously treating the second intermediate product to separate the fatty acid methyl esters from the glycerin to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

5

10

15

20

continuously purifying the fatty acid methyl ester rich stream and recovering methanol from it to produce a purified biodiesel product and a first wet methanol stream;

continuously purifying the glycerin rich stream to produce a purified glycerin product and a second wet methanol stream;

continuously purifying the wet methanol streams to produce a purified methanol product; and

recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acids; and

recycling at least a portion of the purified methanol product into the transesterification reactor for reaction with the glycerides.

Claim 50. The process of claim 49 wherein the feedstock comprises an animal fat and/or a vegetable oil.

Claim 51. The process of claim 49 wherein the effective amount of glycerin is about two times the stoichiometric amount of fatty acids in the feedstock.

Claim 52. The process of claim 49 wherein the reacting step is carried out at a temperature in the range of about 200°C to about 250°C, under agitation and to the extent that the first intermediate product contains no more than 0.5 percent w/w of free fatty acids.

Claim 53. The process of claim 49 wherein the performing step comprises adding potassium methoxide to the intermediate product to facilitate base catalysis and wherein the performing step is carried out at a temperature in the range of about 40°C to about 60°C.

Claim 54. A system for the production of biodiesel from a lipid feedstock comprising a plurality of free fatty acids, said system comprising:

means for introducing the lipid feedstock to a conditioning reactor;

means for conditioning the lipid feedstock in the conditioning reactor, said means for conditioning comprising

means for heating the lipid feedstock,
an acoustic transducer or an impellor system, and
means for filtering the lipid feedstock comprising a traveling screen,
said means for conditioning being operative to produce a conditioned lipid

feedstock;

5

10

15

20

means for measuring the concentration of free fatty acids in the conditioned lipid feedstock;

first means for reacting the plurality of free fatty acids in the conditioned lipid feedstock with a purified glycerin product in a glycerolysis reactor, said first means for reacting comprising

first means for mixing the plurality of free fatty acids in the feedstock with the purified glycerin product, said first means for mixing comprising an acoustic transducer or an impellor system, and

means for continuously reacting the plurality of fatty acids with the purified glycerin product in the absence of a catalyst in a glycerolysis reaction,

5

10

15

20

means for continuously adding the purified glycerin product to the glycerolysis reactor, and

means for removing water continuously from the glycerolysis reactor as a vapor, said means for removing water comprising a fractionation column that is operative to return condensed glycerin to the glycerolysis reactor,

said glycerolysis reactor comprising at least two continuous stirred tank reactors that are operated in series,

said at least two continuous stirred tank reactors having a combined residence time of about 200 minutes,

said first means for reacting being operative to produce a glycerolysis reactor effluent stream;

second means for reacting the plurality of glycerides contained in the glycerolysis effluent stream with a purified methanol product in a transesterification reactor, said second means for reacting comprising

second means for mixing the plurality of glycerides with said purified methanol product and potassium hydroxide, said second means for mixing comprising an acoustic transducer or an impellor system,

means for continuously reacting the glycerides with the methanol in an alkali catalyzed transesterification reaction,

5

10

15

20

means for adding the purified methanol product to the transesterification reactor,

means for adding the potassium hydroxide to the transesterification reactor,

said transesterification reactor comprising at least two continuous stirred tank

reactors that are operated in series, said reactors having a combined residence time of about 60

minutes,

said second means for reacting being operative to produce a transesterification reactor effluent stream that contains a plurality of fatty acid methyl esters and glycerin;

means for separating the plurality of fatty acid methyl esters from the glycerin in the transesterification effluent stream, said means for separating comprising

means for concentrating a first liquid phase in which the plurality of fatty acid methyl esters are concentrated and a second liquid phase in which glycerin is concentrated, said means for concentrating comprising a continuous clarifier,

said means for separating being operative to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

first means for purifying the fatty acid methyl ester rich stream comprising a fatty acid methyl ester distillation column and means for recovering methanol from it to produce a purified biodiesel product and a first wet methanol stream;

second means for purifying the glycerin rich stream and recovering methanol from it to produce the purified glycerin product and a second wet methanol stream, said second means for purifying comprising

means for reacting the potassium hydroxide in the glycerin rich stream with phosphoric acid to produce an insoluble salt having fertilizer value,

a solids separation operation for removing the insoluble salt from the glycerin rich stream,

means for rinsing the insoluble salt with methanol and filtering the insoluble salt, means for adjusting the pH of the glycerin rich stream to about neutral by adding an alkali solution and

a glycerin distillation column for further purifying the glycerin rich stream and a decolorization column comprising a packed bed of activated carbon column;

third means for purifying the wet methanol streams by removing water to produce a purified methanol product, said third means for purifying comprising a methanol distillation column wherein the wet methanol streams are purified;

first means for recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the plurality of free fatty acids; and

second means for recycling at least a portion of the purified methanol product into the transesterification reactor for reaction with the plurality of glycerides.

15

5

10

Claim 55. A system for the production of biodiesel from a lipid feedstock comprising a free fatty acid, said system comprising:

means for conditioning the lipid feedstock in a conditioning reactor, said means for conditioning being operative to heat, mix and filter the lipid feedstock to produce a conditioned lipid feedstock;

first means for reacting the free fatty acid in the conditioned lipid feedstock with a purified glycerin product in a glycerolysis reactor, said first means for reacting comprising

5

10

15

20

first means for mixing the free fatty acid in the feedstock, and
means for continuously reacting the free fatty acid with the purified glycerin
product in the absence of a catalyst in a glycerolysis reaction,

means for continuously adding the purified glycerin product to the glycerolysis reactor at a rate that is greater than the stoichiometric amount of glycerin required for the glycerolysis reaction, and

means for continuously removing water from the glycerolysis reactor as a vapor, said first means for reacting being operative to produce a glycerolysis reactor effluent stream that contains a glyceride;

second means for reacting the glyceride contained in the glycerolysis effluent stream with a purified alcohol product in a transesterification reactor, said second means for reacting comprising

second means for mixing the glyceride with said purified alcohol product,
means for continuously reacting the glyceride with the alcohol in an alkali
catalyzed transesterification reaction,

means for adding the purified alcohol product to the transesterification reactor at a rate that is greater than the stoichiometric amount of alcohol required for the alkali catalyzed transesterification reaction,

said second means for reacting being operative to produce a transesterification reactor effluent stream that contains a fatty acid methyl ester and glycerin;

means for separating the plurality of fatty acid methyl esters from the glycerin in the transesterification effluent stream, said means for separating comprising a continuous operation, and said means for separating being operative to produce a first liquid phase in which the plurality of fatty acid methyl esters are concentrated and a second liquid phase in which glycerin is concentrated to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

first means for purifying the fatty acid methyl ester rich stream comprising a fatty acid methyl ester distillation column or a fatty acid methyl ester fractionation column and first means for recovering alcohol from the fatty acid methyl ester rich stream that are operative to produce a purified biodiesel product and a first wet alcohol stream;

second means for purifying the glycerin rich stream and second means for recovering alcohol from the glycerin rich stream that are operative to produce the purified glycerin product and a second wet alcohol stream, said second means for purifying comprising

means for reacting the alkali in the glycerin rich stream with an acid to produce an insoluble salt,

means for removing the insoluble salt from the glycerin rich stream,

means for filtering the insoluble salt and rinsing the insoluble salt with the
alcohol,

92

15

20

5

10

means for adjusting the pH of the glycerin rich stream to about neutral, a glycerin distillation column or a glycerin fractionation column, and a decolorization column; and

third means for purifying the wet alcohol streams by removing water that is operative to produce a purified alcohol product, said third means for purifying comprising an alcohol distillation column or an alcohol fractionation column.

Claim 56. The system of claim 55 further comprising:

first means for recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acid.

Claim 57. The system of claim 56 further comprising:

second means for recycling at least a portion of the purified alcohol product into the transesterification reactor for reaction with the glyceride.

15

10

5

Claim 58. The system of claim 57 further comprising means for delivering at least a portion of the purified biodiesel product and at least a portion of the purified glycerin product to their markets.

Claim 59. A system for the production of biodiesel from a lipid feedstock comprising a free fatty acid, said system comprising:

means for continuously conditioning the lipid feedstock that is operative to produce a conditioned lipid feedstock;

means for continuously measuring the concentration of the free fatty acid in the conditioned lipid feedstock;

5

10

15

20

first means for continuously reacting the free fatty acid in the conditioned lipid feedstock in a glycerolysis reaction that is operative to react the free fatty acid in the feedstock with glycerin to produce a glyceride;

second means for continuously reacting the glyceride in a transesterification reaction that is operative to convert the glyceride to a fatty acid methyl ester and glycerin via an alkali catalyzed reaction;

means for continuously separating the fatty acid methyl ester from the glycerin that is operative to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

first means for continuously purifying the fatty acid methyl ester rich stream and means for recovering methanol from the fatty acid methyl ester rich stream that are operative to produce a purified biodiesel product and a first wet methanol stream;

second means for continuously purifying the glycerin rich stream that is operative to produce a purified glycerin product and a second wet methanol stream;

third means for continuously purifying the wet methanol streams that is operative to produce a purified methanol product; and

first means for recycling at least a portion of the purified glycerin product into the first means for continuously reacting for reaction with the free fatty acid.

Claim 60. The system of claim 59 further comprising:

means for recycling at least a portion of the purified methanol product into the second means for continuously reacting for reaction with the glyceride.

Claim 61. A system for the production of biodiesel from a lipid feedstock comprising a free fatty acid, said system comprising:

means for introducing a lipid feedstock to a conditioning reactor;

5

10

15

20

means for conditioning the lipid feedstock that is operative to heat, mix and filter the lipid feedstock to produce a conditioned lipid feedstock;

first means for reacting the free fatty acid in the conditioned lipid feedstock comprising a glycerolysis reactor that is operative to continuously react the free fatty acid in the feedstock with glycerin to produce a glyceride via a glycerolysis reaction;

second means for reacting the glyceride comprising a transesterification reactor that is operative to continuously convert the glyceride to a fatty acid methyl ester and glycerin via an alkali catalyzed transesterification reaction;

means for separating the fatty acid methyl ester from the glycerin that is operative to produce a first liquid phase in which the fatty acid methyl ester is concentrated and a second liquid phase in which glycerin is concentrated that are continuously separated to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

first means for purifying the fatty acid methyl ester rich stream and first means for recovering methanol from the fatty acid methyl ester rich stream that are operative to produce a purified biodiesel product and a first wet methanol stream;

second means for purifying the glycerin rich stream and means for recovering methanol from the glycerin rich stream that are operative to produce a purified glycerin product and a second wet methanol stream;

third means for purifying the wet methanol streams by removing water from them that is operative to produce a purified methanol product;

first means for recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acid; and

second means for recycling at least a portion of the purified methanol product into the transesterification reactor for reaction with the glyceride.

10

5

Claim 62. A system for the production of biodiesel from a lipid feedstock comprising a free fatty acid, said system comprising:

a conditioning reactor that is operative to heat, mix and filter the lipid feedstock to produce a conditioned lipid feedstock;

15

an in-line free fatty acid titration device that is operative to quantify the concentration of the free fatty acid in the conditioned lipid feedstock;

a continuous glycerolysis reactor that is operative to continuously react the free fatty acid in the conditioned lipid feedstock with glycerin to produce a glyceride via a glycerolysis reaction in response to a signal from the in-line free fatty acid titration device;

20

a continuous transesterification reactor that is operative to continuously convert the glyceride to a fatty acid methyl ester and glycerin via an alkali catalyzed transesterification reaction in response to the signal from the in-line free fatty acid titration device;

a phase separation centrifuge that is operative to produce a first liquid phase in which the fatty acid methyl ester is concentrated and a second liquid phase in which glycerin is concentrated that are continuously separated to produce a fatty acid methyl ester rich stream and a glycerin rich stream;

5

a fatty acid methyl ester fractionation column that is operative to produce a purified biodiesel product and a first wet methanol stream;

a glycerin fractionation column that is operative to produce a purified glycerin product and a second wet methanol stream;

a methanol fractionation column that is operative to treat the first wet methanol stream and the second wet methanol stream to produce a purified methanol product;

first pump for recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acid; and

second pump for recycling at least a portion of the purified methanol product into the transesterification reactor for reaction with the glyceride.

15

10

Claim 63. A process for the production of biodiesel from a lipid feedstock comprising a plurality of free fatty acids, said process comprising:

20

reacting the plurality of free fatty acids in the lipid feedstock with a purified glycerin product in a glycerolysis reactor, wherein the plurality of free fatty acids in the feedstock is mixed with the purified glycerin product in an agitated vessel to produce a glycerolysis reactor effluent stream that contains less than 0.5 percent by weight of free fatty acids and a plurality of glycerides, the purified glycerin product being added to the glycerolysis reactor in the range of about 110 percent to about 400 percent of the stoichiometric amount of glycerin required for the

glycerolysis reaction, and water being continuously removed from the glycerolysis reactor as a vapor through a vent in the reactor headspace;

reacting the plurality of glycerides contained in the glycerolysis effluent stream with an alcohol in a transesterification reactor, wherein the plurality of glycerides are mixed with said alcohol and a catalyst to produce a transesterification reactor effluent stream that contains a plurality of fatty acid alkyl esters and glycerin, the alcohol being added to the transesterification reactor as a purified alcohol product at a rate equal to about 200 percent of the stoichiometric amount of alcohol required for the catalyzed reaction, and the catalyst being added to the transesterification reactor at a rate sufficient to catalyze the reaction;

10

5

separating the plurality of fatty acid alkyl esters from the glycerin in the transesterification effluent stream by the difference in their densities, wherein the force of gravity or centrifugal force is used to separate two distinct immiscible phases, a first liquid phase in which the plurality of fatty acid alkyl esters are concentrated and a second liquid phase in which glycerin is concentrated, to produce a fatty acid alkyl ester rich stream and a glycerin rich stream;

15

purifying the fatty acid alkyl ester rich stream by using the differences in the vapor pressures of the components of the fatty acid alkyl ester rich stream, and recovering a first alcohol stream, a high purity biodiesel product and a first high boiling point impurities stream;

20

purifying the glycerin rich stream and recovering a second alcohol stream, the purified glycerin product and a second high boiling point impurities stream;

purifying the recovered alcohol streams by removing water from them to produce the purified alcohol product;

recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acids in the feedstock; and

recycling at least a portion of the purified alcohol product into the transesterification operation for reaction with the plurality of glycerides.

5

10

15

Claim 64. The process of claim 63 further comprising:

prior to the step of reacting the plurality of free fatty acids, conditioning the feedstock by elevating the temperature of the feedstock to at least 35°C, mixing the feedstock and filtering out insoluble solids to generate a uniform mixture of glycerides, free fatty acids and unsaponafiable materials.

Claim 65. The process of claim 63 wherein the glycerolysis reactor is a continuous reactor and the step of reacting the plurality of free fatty acids is carried out in a continuous fashion.

Claim 66. The process of claim 65 wherein the step of reacting the plurality of free fatty acids is carried out in a series of at least two continuous stirred tank reactors.

Claim 67. The process of claim 65 wherein the step of reacting the plurality of free fatty acids is carried out without a catalyst at a temperature between 150°C and 250°C and at a pressure between 0.1 pounds per square inch absolute and 7.0 pounds per square inch absolute.

20

Claim 68. The process of claim 66 wherein the vapor vented from the glycerolysis reactor is fractionated to yield a liquid fraction having a high concentration of glycerin and a vapor fraction

having a high concentration of water, the liquid fraction being recycled to the glycerolysis reactor.

Claim 69. The process of claim 63 wherein the transesterification reactor is a continuous reactor and the step of reacting the plurality of glycerides is carried out in a continuous fashion.

5

10

15

20

Claim 70. The process of claim 69 wherein the step of reacting the plurality of glycerides is carried out in a plug flow reactor.

Claim 71. The process of claim 69 wherein the step of reacting the plurality of glycerides is carried out in a series of at least two continuous stirred tank reactors.

Claim 72. The process of claim 63 wherein the step of reacting the plurality of glycerides is carried out in the presence of a caustic alkali catalyst selected from the group consisting of lithium hydroxide, sodium hydroxide and potassium hydroxide.

Claim 73. The process of claim 63 wherein at least a portion of the glycerin is removed from the transesterification reactor before the step of reacting the plurality of glycerides is complete.

Claim 73. The process of claim 63 wherein multiple alcohol or catalyst additions are made to the transesterification reactor.

Claim 75. A process for the production of biodiesel from a feedstock comprising a glyceride, said process comprising:

reacting the glyceride in the feedstock with a purified alcohol product comprising an alcohol in a transesterification reactor, wherein the glyceride is mixed with said purified alcohol product and continuously reacted with the alcohol at an appropriate temperature and pressure in an alkali catalyzed transesterification reaction to produce a transesterification reactor effluent stream that contains a fatty acid alkyl ester and glycerin, the purified alcohol product being added to the transesterification reactor at a rate that is greater than the stoichiometric amount of alcohol required for the alkali catalyzed transesterification reaction;

10

5

separating the plurality of fatty acid alkyl esters from the glycerin in the transesterification effluent stream in continuous operation, wherein a first liquid phase in which the plurality of fatty acid alkyl esters are concentrated and a second liquid phase in which glycerin is concentrated are continuously separated to produce a fatty acid alkyl ester rich stream and a glycerin rich stream;

15

purifying the fatty acid alkyl ester rich stream in a fatty acid alkyl ester distillation column or a fatty acid alkyl ester fractionation column and recovering the alcohol from it to produce a purified biodiesel product and a first wet alcohol stream;

20

purifying the glycerin rich stream and recovering the alcohol from it to produce the purified glycerin product and a second wet alcohol stream, wherein the alkali in the glycerin rich stream is reacted with an acid to produce an insoluble salt that is removed from the glycerin rich stream and thereafter filtered and rinsed with the alcohol, the pH of the glycerin rich stream being adjusted to neutral and the glycerin rich stream being further purified in a glycerin distillation column or a glycerin fractionation column and in a decolorization column;

purifying the wet alcohol streams by removing water to produce a purified alcohol product, wherein the wet alcohol streams are purified in an alcohol distillation column or an alcohol fractionation column; and

recycling at least a portion of the purified alcohol product into the transesterification reactor for reaction with the glyceride.

5

10

15

20

Claim 76. A process for the production of biodiesel from a lipid feedstock comprising a plurality of free fatty acids, said process comprising:

reacting the plurality of free fatty acids in the lipid feedstock with a purified glycerin product in a glycerolysis reactor, wherein the plurality of free fatty acids in the feedstock is mixed with the purified glycerin product in an agitated vessel to produce a glycerolysis reactor effluent stream that contains less than 0.5 percent by weight of free fatty acids and a plurality of glycerides, the purified glycerin product being added to the glycerolysis reactor in the range of about 110 percent to about 400 percent of the stoichiometric amount of glycerin required for the glycerolysis reaction, and water being continuously removed from the glycerolysis reactor as a vapor through a vent in the reactor headspace;

reacting the plurality of glycerides contained in the glycerolysis effluent stream with an alcohol in a transesterification reactor, wherein the plurality of glycerides are mixed with said alcohol and a catalyst to produce a transesterification reactor effluent stream that contains a plurality of fatty acid alkyl esters and glycerin, the alcohol being added to the transesterification reactor as a purified alcohol product at a rate equal to about 200 percent of the stoichiometric amount of alcohol required for the catalyzed reaction, and the catalyst being added to the transesterification reactor at a rate sufficient to catalyze the reaction;

separating the plurality of fatty acid alkyl esters from the glycerin in the transesterification effluent stream by means of membrane filtration into two distinct immiscible phases, a first liquid phase in which the plurality of fatty acid alkyl esters are concentrated and a second liquid phase in which glycerin is concentrated, to produce a fatty acid alkyl ester rich stream and a glycerin rich stream;

(

5

10

15

purifying the fatty acid alkyl ester rich stream by using the differences in the vapor pressures of the components of the fatty acid alkyl ester rich stream, and recovering a first alcohol stream, a high purity biodiesel product and a first high boiling point impurities stream;

purifying the glycerin rich stream and recovering a second alcohol stream, the purified glycerin product and a second high boiling point impurities stream;

purifying the recovered alcohol streams by removing water from them to produce the purified alcohol product;

recycling at least a portion of the purified glycerin product into the glycerolysis reactor for reaction with the free fatty acids in the feedstock; and

recycling at least a portion of the purified alcohol product into the transesterification operation for reaction with the plurality of glycerides.